Electronic Supplementary Material (ESI) for Green Chemistry.

Cyrene™ Blends: A Greener Solvent System for Organic Syntheses

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1. General

All reagents and solvents were obtained from MilliporeSigma, unless otherwise noted, and used as received. **As a precaution, heating Cyrene™ and Cyrene™ blends with inorganic base, such as K_2CO_3, KOH, etc., to over 100 °C for long period of time is not recommended without any detailed thermal stability investigation.**

1.1 Preparation of solutions

Cyrene™ blends were prepared by weight in 100 mL screw cap amber glass bottles and mixed thoroughly.

Table S1. Cyrene™ blends and their weight percentage

Cyrene™ blends	**Weight percent**
Cyrene™/γ-Valerolactone	50/50
Cyrene™/2-MeTHF	80/20
Cyrene™/Acetonitrile	80/20
Cyrene™/DMF	80/20

1.2 Experimental details

Reactions were carried out using disposable 20 mL screw cap glass vials or in 20 mL Radley reaction tubes (heated reactions and reactions sensitive to stir rate). Reactions at elevated temperatures were carried out using a Radley reaction station hotplate/stirrer.

1.3 Purification of products

Normal phase flash chromatography was carried out using a Biotage Isolera One. Purification not sensitive to elution rates was carried out using a silica plug (30 g silica gel, 60 mL fritter funnel).

1.4 Analysis of products

^{1}H NMR spectra were obtained on a Bruker Avance Neo spectrometer (RT BBOF smartprobe) at 500 MHz. ^{1}H and ^{19}F NMR spectra were obtained on an Agilent VNMRS (OneNMR BBO probe) at 500 MHz and 470 MHz, respectively. Chemical shifts are reported in ppm and coupling constants are reported in Hz with $CDCl_3$ referenced at 7.26 (^{1}H).

2. Stability Study

2.1 Preparation of solutions

Cyrene™ (neat) was transferred into eight 100 mL screw cap amber glass bottles, labelled “a” through “h”, where “a” through “c” received screw caps, and “d” through “h” received sure-seals and screw caps. Cyrene™ blend mixtures (eight of each mixture) were prepared by weight at the ratios listed in section **1.1** in 100 mL screw cap amber glass bottles and labelled “a” through “h”, where “a” through “e” received screw caps, and “f” through “h” received sure-seals and screw

caps. Cyrene™ (neat) bottles “a” through “e” and Cyrene™ blend mixture bottles “a” through “e” were maintained at ambient temperature, while Cyrene™ (neat) bottles “f” through “h” and Cyrene™ blend mixture bottles “f” through “h” were maintained in an oven at an elevated temperature of 40°C.

2.2 Analysis of solutions

At each pre-determined time, stated in sections **2.3** and **2.4**, each Cyrene™ (neat) bottle and Cyrene™ blend mixture bottle was submitted for the following tests: GC, KF, and ^{1}H NMR. Cyrene™ blend mixture bottles containing 2-MeTHF were submitted for additional testing: peroxides (strip testing; pass/fail) and BHT content. In the sections below (**2.3** and **2.4** for the room temperature and accelerated studies, respectively) each Cyrene™ (neat) and Cyrene™ blend mixture bottle was given a “pass”/“fail” based on the amalgamation of all above stated tests. Cyrene™ (neat) bottle “a” and Cyrene™ blend mixture bottles “a” were submitted for initial DSC testing, as well as the above stated tests (see section **2.3**).

Table S2. Boiling point (DSC, °C) of Cyrene™ blends in the study.

Solvent	**DSC (b.p., °C)**
Cyrene™	225.52
Cyrene™/γ-Valerolactone (50/50)	216.02
Cyrene™/2-MeTHF (80/20)	228.39
Cyrene™/Acetonitrile (80/20)	229.07
Cyrene™/DMF (80/20)	223.88

2.3 Room temperature study

Cyrene™ (neat) bottles “a” through “e” and Cyrene™ blend mixture bottles “a” through “e” were maintained with caps at ambient temperature until testing.

Table S3. Room temperature stability studies of Cyrene™ blends

Time (months)	**Bottle**	**Cyrene™**	**Cyrene™ GVL**	**Cyrene™ 2-MeTHF**	**Cyrene™ CH_3CN**	**Cyrene™ DMF**
0	a	Pass	Pass	Pass	Pass	Pass
3	b	Pass	Pass	Pass	Pass	Pass
6	c	Pass	Pass	Pass	Pass	Pass
12	d	Pass	Pass	Pass	Pass	Pass

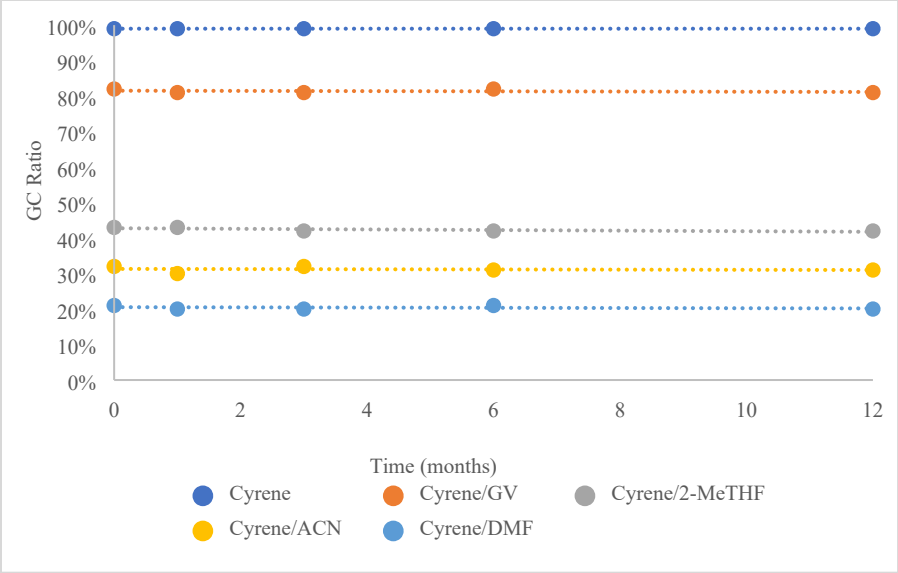


Figure S1. Room temperature stability studies on Cyrene™ blends

2.4 Accelerated study at temperature of 40 °C

Cyrene™ (neat) bottles "f" through "h" and Cyrene™ blend mixture bottles "f" through "h" were maintained with sure-seals and caps in an oven at an elevated temperature of 40°C until testing.

Table S4. Elevated temperature (40°C) stability studies of Cyrene™ blends

Time (weeks)	Bottle	Cyrene™	Cyrene™ GVL	Cyrene™ 2-MeTHF	Cyrene™ CH_3CN	Cyrene™ DMF
6	f	Pass	Pass	Pass	Pass	Pass
12	g	Pass	Pass	Pass	Pass	Pass
24	h	Pass	Pass	Pass	Pass	Pass

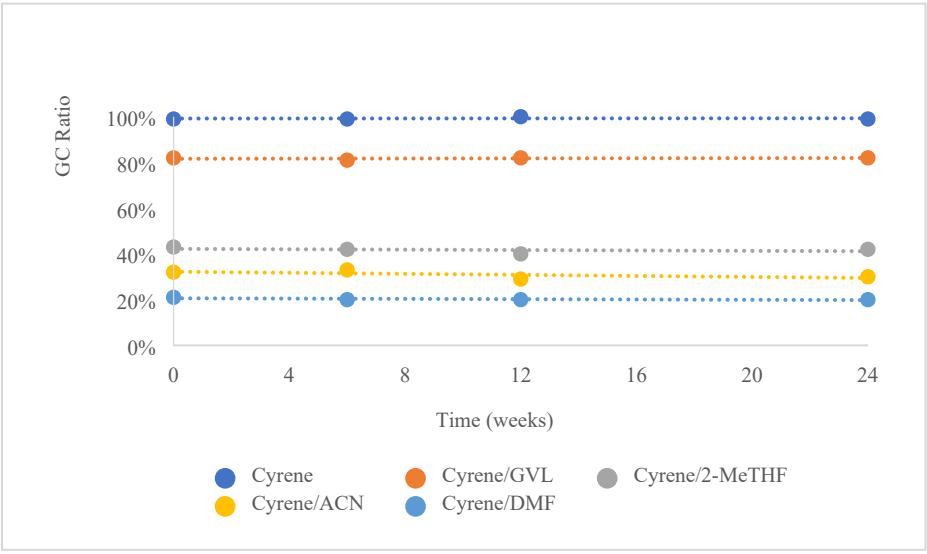


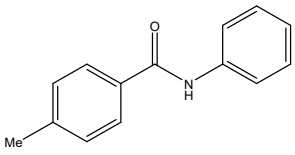
Figure S2. Elevated temperature (40 °C) stability studies of Cyrene™ blends

3. Application Studies in Organic Syntheses

3.1 Amide Coupling

3.1.1 Synthesis of 4-methyl-*N*-phenylbenzamide (1)

Procedure derived from an amide cross-coupling conducted using Cyrene™.[1]



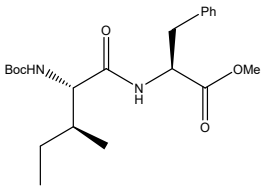
To a 20 mL Radley reaction tube was added *p*-toluic acid (170 mg, 1.25 mmol, 1 equiv.), HATU (570 mg, 1.50 mmol, 1.2 equiv.), *N,N*-diisopropylethylamine (655 µL, 3.76 mmol, 3 equiv.), and **solvent** (6.25 mL). The reaction mixture was stirred at 800 rpm for 30 minutes before the addition of aniline (125 µL, 1.37 mmol, 1.1 equiv.) and subsequently maintained at room temperature for 2 hours while stirring at 800 rpm. The solution was then diluted with EtOAc (10 mL) and washed with 1 M HCl (2 x 20 mL) and brine (20 mL). The organics were then dried over Na_2SO_4 and concentrated under reduced pressure to give a residue which was subsequently purified by flash chromatography (silica gel, 0-20% EtOAc in petroleum ether) to afford the title compound as a white solid. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.79 (s, 1H), 7.78 – 7.75 (m, 2H), 7.66 – 7.62 (m, 2H), 7.40 – 7.35 (m, 2H), 7.31 – 7.27 (m, 2H), 7.18 – 7.12 (m, 1H), 2.43 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 165.67, 142.39, 138.06, 132.14, 129.46, 129.09, 127.04, 124.44, 120.17, 21.51.

Table S5. Isolated yields of product **1**

Entry	Solvent	Isolated Yield
1	Cyrene™	80.4%
2	Cyrene™/γ-Valerolactone (50/50)	90.7%
3	Cyrene™/2-MeTHF (80/20)	90.2%
4	Cyrene™/Acetonitrile (80/20)	93.9%
5	Cyrene™/DMF (80/20)	81.8%
6	DMF	94.8%
7	NMP	92.6%

3.1.2 Synthesis of Boc-Ile-Phe-OMe (2)

Procedure derived from an amide cross-coupling conducted using Cyrene[1].



To a 20 mL screw cap glass vial was added Boc-Ile-OH (289 mg, 1.25 mmol, 1 equiv.), HATU (570 mg, 1.50 mmol, 1.2 equiv.), *N,N*-diisopropylethylamine (655 µL, 3.76 mmol, 3 equiv.), and **solvent** (6.25 mL). The reaction mixture was stirred for 30 minutes before the addition of Phe-OMe·HCl (296.5 mg, 1.37 mmol, 1.1 equiv.) and subsequently maintained at room temperature for 2 hours while stirring. The solution was then diluted with EtOAc (10 mL) and washed with 1 M HCl (2 x 20 mL) and brine (20 mL). The organics were then dried over Na_2SO_4 and concentrated under reduced pressure to give a residue which was subsequently purified by silica plug (30 g silica gel, eluted in 100% EtOAc) to afford the title compound as a white solid. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.31 – 7.27 (m, 2H), 7.26 – 7.22 (m, 1H), 7.13 – 7.10 (m, 2H), 6.29 (d, *J* = 7.7 Hz, 1H), 4.99 (d, *J* = 7.6 Hz, 1H), 4.87 (dt, *J* = 7.9, 5.9 Hz, 1H), 3.96 – 3.89 (m, 1H), 3.71 (s, 3H), 3.12 (qd, *J* = 13.9, 5.9 Hz, 2H), 1.82 (dt, *J* = 9.9, 5.1 Hz, 1H), 1.44 (s, 9H), 0.87 (dd, *J* = 11.0, 6.8 Hz, 6H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.66, 171.19, 135.67, 129.26, 128.63, 127.19, 59.26, 53.08, 52.32, 38.00, 37.23, 28.32, 24.66, 15.43, 11.44.

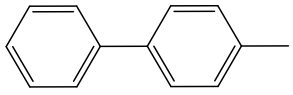
Table S6. Isolated yields of product **2**

Entry	Solvent	Isolated Yield
1	Cyrene™	97.9%
2	Cyrene™/γ-Valerolactone (50/50)	96.0%
3	Cyrene™/2-MeTHF (80/20)	96.3%
4	Cyrene™/Acetonitrile (80/20)	98.2%
5	Cyrene™/DMF (80/20)	81.1%
6	DMF	98.9%
7	NMP	98.6%

3.2 Suzuki coupling

3.2.1 Synthesis of 4-phenyltoluene (3)

Procedure derived from a palladium-catalyzed Suzuki-Miyaura cross-coupling conducted using Cyrene™.[2]



To a 20 mL Radley reaction tube was added Pd(dppf)Cl_2·CH_2Cl_2 (41.0 mg, 0.05 mmol, 0.04 equiv.), 4-bromotoluene (214 mg, 1.25 mmol, 1 equiv.), Cs_2CO_3 (1.2225 g, 3.75 mmol, 3 equiv.), phenylboronic acid (228.7 mg, 1.88 mmol, 1.5 equiv.) or phenylboronic acid MIDA ester (437.2 mg, 1.88 mmol, 1.5 equiv.), **solvent** (5 mL), and water (9 mL, 400 equiv.). The tube was then capped and placed under N_2. The reaction mixture was heated to 50°C with stirring at 800 rpm

and maintained at this temperature and stir rate for 16 hours before the vessel was vented and decapped. The solution was then diluted with EtOAc (10 mL) and washed with water (2 x 20 mL) and brine (2 x 20 mL). The organics were then dried over Na_2SO_4 and concentrated under reduced pressure to give a yellow oil which was purified by silica plug (30 g silica gel, eluted in 100% petroleum ether) to afford the title compound as a white solid. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.63 – 7.59 (m, 2H), 7.54 – 7.51 (m, 2H), 7.48 – 7.43 (m, 2H), 7.38 – 7.33 (m, 1H), 7.30 – 7.27 (m, 2H), 2.43 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 141.22, 138.41, 137.06, 129.52, 128.80, 128.76, 127.02, 127.01, 21.14.

Table S7. Isolated yield of product **3** from phenylboronic acid

Entry	Solvent	Isolated Yield
1	Cyrene™	82%
2	Cyrene™/γ-Valerolactone (50/50)	95%
3	Cyrene™/2-MeTHF (80/20)	91%
4	Cyrene™/Acetonitrile (80/20)	72%
5	Cyrene™/DMF (80/20)	96%
6	DMF	94%
7	NMP	91%

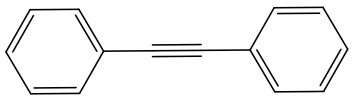
Table S8. Isolated yield of product **3** from phenylboronic acid MIDA ester

Entry	Solvent	Isolated Yield
1	Cyrene™	78%
2	Cyrene™/γ-Valerolactone (50/50)	85%
3	Cyrene™/2-MeTHF (80/20)	80%
4	Cyrene™/Acetonitrile (80/20)	88%
5	Cyrene™/DMF (80/20)	92%
6	DMF	87%
7	NMP	90%

3.3 Sonogashira Coupling

3.3.1 Synthesis of diphenylacetylene (4)

Procedure derived from a palladium-catalyzed Sonogashira cross-coupling conducted using Cyrene™. [3]



To a 20 mL Radley reaction tube was added $Pd(PPh_3)_2Cl_2$ (17.5 mg, 0.02 mmol, 0.02 equiv.), CuI (9.5 mg, 0.05 mmol, 0.04 equiv.), iodobenzene (139.5 µL, 1.25 mmol, 1 equiv.), phenylacetylene (178 µL, 1.62 mmol, 1.3 equiv.), Et_3N (190 µL, 1.36 mmol, 1.1 equiv.), and **solvent** (2.5 mL). The tube was then capped and placed under N_2. The reaction mixture was heated to 30°C with stirring

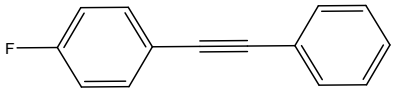
at 800 rpm and maintained at this temperature and stir rate for 2 hours before the vessel was vented and decapped. The solution was then diluted with EtOAc (5 mL) and washed with water (2 x 20 mL) and brine (2 x 20 mL). The organics were then dried over $MgSO_4$ and concentrated under reduced pressure to give a yellow oil which was purified by silica plug (30 g silica gel, eluted in 100% petroleum ether) to afford the title compound as a white solid. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.58 – 7.50 (m, 4H), 7.38 – 7.31 (m, 6H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 131.76, 128.49, 128.40, 123.43, 89.52.

Table S9. Isolated yields of Product **4**

Entry	Solvent	Isolated Yield
1	Cyrene™	94%
2	Cyrene™/γ-Valerolactone (50/50)	92%
3	Cyrene™/2-MeTHF (80/20)	89%
4	Cyrene™/Acetonitrile (80/20)	86%
5	Cyrene™/DMF (80/20)	93%
6	DMF	97%
7	NMP	96%

3.3.2 Synthesis of 1-fluoro-4-(phenylethynyl)benzene (5)

Procedure derived from a palladium-catalyzed Sonogashira cross-coupling conducted using Cyrene™. [3]



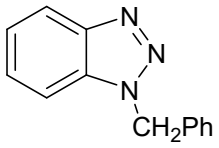
To a 20 mL Radley reaction tube was added $Pd(PPh_3)_2Cl_2$ (17.5 mg, 0.02 mmol, 0.02 equiv.), CuI (9.5 mg, 0.05 mmol, 0.04 equiv.), 4-fluoroiodobenzene (144 µL, 1.25 mmol, 1 equiv.), phenylacetylene (178 µL, 1.62 mmol, 1.3 equiv.), Et_3N (190 µL, 1.36 mmol, 1.1 equiv.), and **solvent** (2.5 mL). The tube was then capped and placed under N_2. The reaction mixture was heated to 30°C with stirring at 800 rpm and maintained at this temperature and stir rate for 2 hours before the vessel was vented and decapped. The solution was then diluted with EtOAc (5 mL) and washed with water (2 x 20 mL) and brine (2 x 20 mL). The organics were then dried over $MgSO_4$ and concentrated under reduced pressure to give a yellow oil which was purified by silica plug (30 g silica gel, eluted in 100% petroleum ether) to afford the title compound as a white solid. Around 5% 1,4-diphenyl-1,3-butadiyne was identified by ^{13}C NMR and GC-MS as an inseparable byproduct (labeled as * in the corresponding spectra).[4] ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.55 – 7.51 (m, 4H), 7.38 – 7.32 (m, 3H), 7.08 – 7.02 (m, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 162.65 (d, J = 249.6 Hz), 133.62 (d, J = 8.4 Hz), 131.71, 128.52, 128.48, 123.24, 119.53 (d, J = 3.5 Hz), 115.79 (d, J = 22.1 Hz), 89.18 (d, J = 1.5 Hz), 88.43. ^{19}F NMR ($CDCl_3$, 470 MHz): δ -111.23 (tt, J = 8.6, 5.4 Hz).

Table S10. Isolated yields of product **5**

Entry	Solvent	Isolated Yield
1	Cyrene™	93%

2	Cyrene™/γ-Valerolactone (50/50)	90%
3	Cyrene™/2-MeTHF (80/20)	81%
4	Cyrene™/Acetonitrile (80/20)	97%
5	Cyrene™/DMF (80/20)	97%
6	DMF	95%
7	NMP	89%

3.4 Synthesis of 1-benzyl-1H-1,2,3-benzotriazole (6)[5]

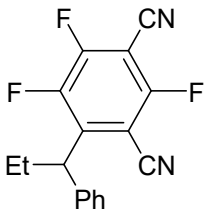


To a 20 mL vial charged with benzotriazole (60 mg, 0.5 mmol) was added solvent (5 mL) followed by benzyl bromide (94 mg, 0.55 mmol) and DBU (84 mg, 0.55 mmol). The resulting mixture was allowed to stir art room temperature for 10 hours. At the conclusion of reaction, the reaction mixture was poured into water (20 mL) and the aqueous solution was extracted by EtOAc (20 mL) for 4 times. Then the organic layers were combined, dried with $MgSO_4$ and evaporated. The resulting residue was further purified through Biotage (EtOAc/Heptane = 5/100 – 30/100) to afford the desired product as a white solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.09 (dt, J = 8.2, 1.1 Hz, 1H), 7.47 – 7.25 (m, 8H), 5.88 (s, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 146.49, 134.89, 132.93, 129.14, 128.60, 127.71, 127.53, 124.04, 120.22, 109.85, 52.40.

Table S11. Isolated yields of product **6**

Entry	**Solvent**	**Isolated Yield**
1	Cyrene™	60%
2	Cyrene™/γ-Valerolactone (50/50)	58%
3	Cyrene™/2-MeTHF (80/20)	66%
4	Cyrene™/Acetonitrile (80/20)	74%
5	Cyrene™/DMF (80/20)	69%
6	DMF	60%
7	NMP	59%

3.5 Synthesis of 3FEPAIPN (7) [6]

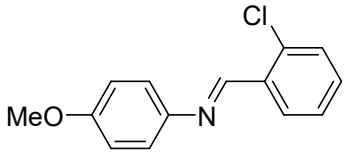


To a 20 mL vial charged with tetrafluoroisophthalonitrile (200 mg, 1.0 mmol) was added solvent (5 mL) followed by N-Ethylaniline (133 mg, 1.1 mmol) and triethylamine (110 mg, 1.1 mmol). The resulting mixture was allowed to stir art room temperature for 4 hours. At the conclusion of reaction, the reaction mixture was poured into water (50 mL) and this aqueous solution was extracted by EtOAc (30 mL) for 4 times. Then the organic layers were combined, dried with $MgSO_4$ and evaporated. The resulting residue was further purified through Biotage (EtOAc/Heptane = 0/100 – 10/100) to afford the desired product as a yellow solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.43 – 7.36 (m, 2H), 7.25 – 7.20 (m, 1H), 7.11 – 7.06 (m, 2H), 4.01 (qd, J = 7.1, 1.7 Hz, 2H), 1.30 (td, J = 7.1, 0.8 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 161.91 (d, *J* = 270.1), 154.36 (ddd, *J* = 268.3, 15.5, 7.7 Hz), 145.24, 144.19, 141.87 (ddd, *J* = 250.6, 12.7, 4.1 Hz), 130.11, 126.01, 122.73, 109.30, 107.52 (d, *J* = 3.0 Hz), 94.36 (d, *J* = 16.7 Hz), 85.97, 49.38 (d, *J* = 6.2 Hz), 13.88 (d, *J* = 4.7 Hz). ^{19}F NMR (470 MHz, $CDCl_3$) δ -99.47 – -101.95 (m), -118.78 – -123.56 (m), -145.51 (dd, *J* = 19.3, 10.6 Hz).

Table S 12. Isolated yields of product **7**

Entry	Solvent	Isolated Yield
1	Cyrene™	44%
2	Cyrene™/γ-Valerolactone (50/50)	46%
3	Cyrene™/2-MeTHF (80/20)	45%
4	Cyrene™/Acetonitrile (80/20)	44%
5	Cyrene™/DMF (80/20)	46%
6	DMF	52%
7	NMP	47%

3.6 Synthesis of N-[(2-chlorophenyl)methylene]-4-methoxy-benzenamine (8)[7]



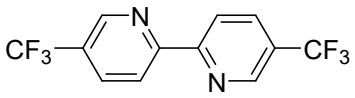
To a 20 mL vial was added p-Anisidine (246 mg, 0.2 mmol) and solvent (4 mL). The mixture was stirred at room temperature until all solid dissolved. 2-Chlorobenzaldehyde (281 mg, 0.2 mmol) was added to the reaction mixture, followed by adding Pyrrolidine (14 mg, 0.02 mmol, 10 mol%). The reaction mixture was stirred at room temperature for 4h. The reaction mixture was then diluted with 10 mL heptane, filtered through a silica gel plug and eluted with 200 mL 10% EtOAc in heptane. The filtrate was collected, and concentrated to yield a yellow residue which was purified using Biotage (EtOAc/Heptane = 0/100 – 10/100). Yellow solid. ^{1}H NMR (CDCl3, 500 MHz): δ 8.97 (s, 1H), 8.28 – 8.25 (m, 1H), 7.45 – 7.44 (m, 1H), 7.42 – 7.37 (m, 2H), 7.32 – 7.29 (m, 2H), 6.99 – 6.97 (m, 2H), 3.87 (s, 3H). ^{13}C NMR ($CDCl_3$, 126 MHz) δ 158.68, 154.76, 144.69, 135.83, 133.51, 131.79, 129.93, 128.39, 127.10, 122.55, 114.45, 55.53.

Table S13. Isolated yields of product **8**

Entry	Solvent	Isolated Yield

1	Cyrene™	77%
2	Cyrene™/γ-Valerolactone (50/50)	72%
3	Cyrene™/2-MeTHF (80/20)	79%
4	Cyrene™/Acetonitrile (80/20)	69%
5	Cyrene™/DMF (80/20)	81%
6	DMF	86%
7	NMP	82%

3.7 Synthesis of 5,5'-Bis(trifluoromethyl)-2,2'-bipyridine (9)[8]

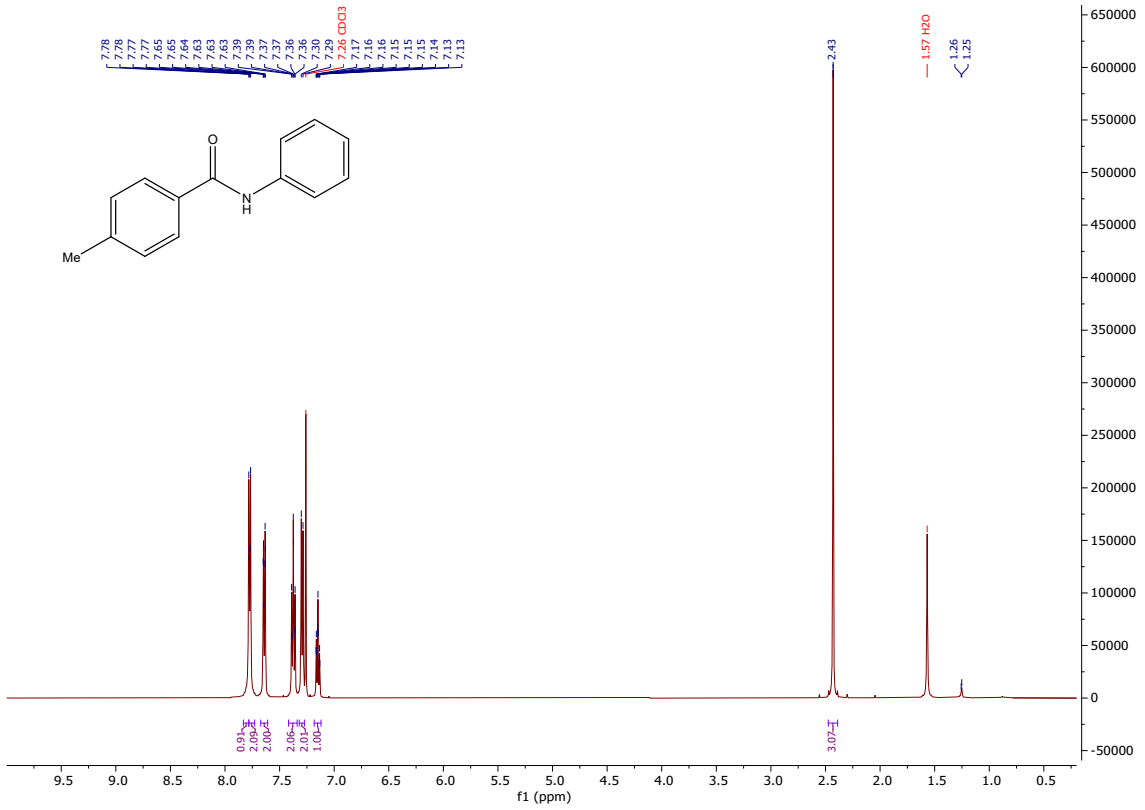


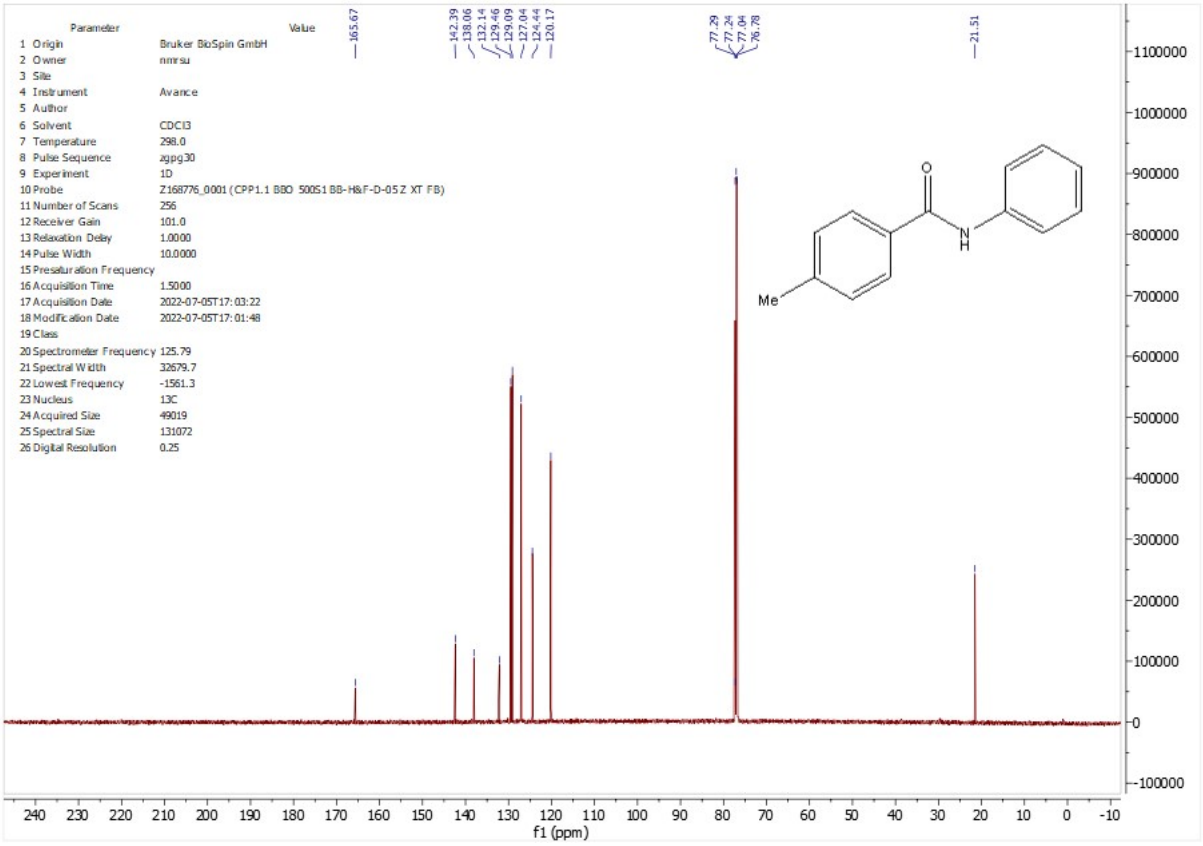
To a 20 mL Radley reaction tube was added 2-bromo-5'-(trifluoromethyl)pyridine (0.75 g, 3.32 mmol, 1 equiv), $Pd(OAc)_2$ (112.5 mg, 0.05 mmol, 15 mol %), TBAI (1.50 g, 4.06 mmol, 1.2 equiv), K_2CO_3 (0.75 g, 5.43 mmol, 1.6 equiv), and solvent (10 mL). The tube was then capped and placed under N_2. The reaction mixture was heated to 50 °C with stirring for about 30 minutes before addition of 2-propanol (0.56 mL, 7.32 mmol, 2.2 equiv). The reaction mixture was maintained at 50 °C for 18 hours. The reaction mixture was cooled down to room temperature, and then filtered through Celite®. The Celite pad was further washed with dichloromethane (20 mL X 2). The filtrate was dried over $MgSO_4$, and concentrated under reduced pressure to give a dark brown oil that was taken up in petroleum ether (3 x 30 mL). The petroleum ether layers were combined and concentrated under reduced pressure to afford the product as a white solid (Cyrene™/2-MeTHF: 50%). ^{1}H NMR ($CDCl_3$, 500 MHz) δ 8.99 (m, 2H), 8.65 (m, 2H), 8.12 (m, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 157.70, 157.69, 146.35, 146.32, 146.29, 146.26, 134.34, 134.31, 134.28, 134.26, 127.54, 127.28, 127.02, 126.75, 124.59, 122.42, 121.25. ^{19}F NMR ($CDCl_3$, 471 MHz) δ –62.69.

5,5'-Bis(trifluoromethyl)-2,2'-bipyridine was also synthesized at various scales (2-bromo-5'-(trifluoromethyl)pyridine/solvent: 5g/20 mL and 50g/200 mL), and isolated with similar yields (ca. 75%).

4. NMR spectra

4-methyl-*N*-phenylbenzamide (1)

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5 Author
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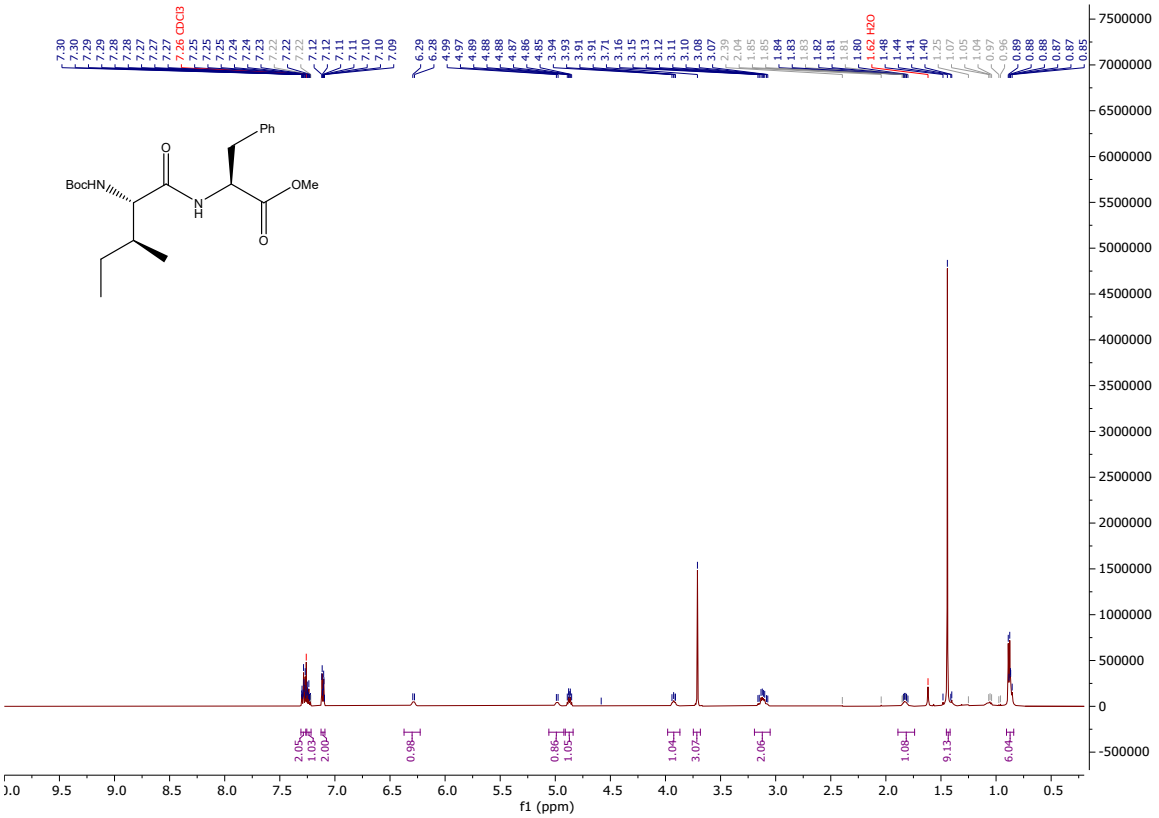
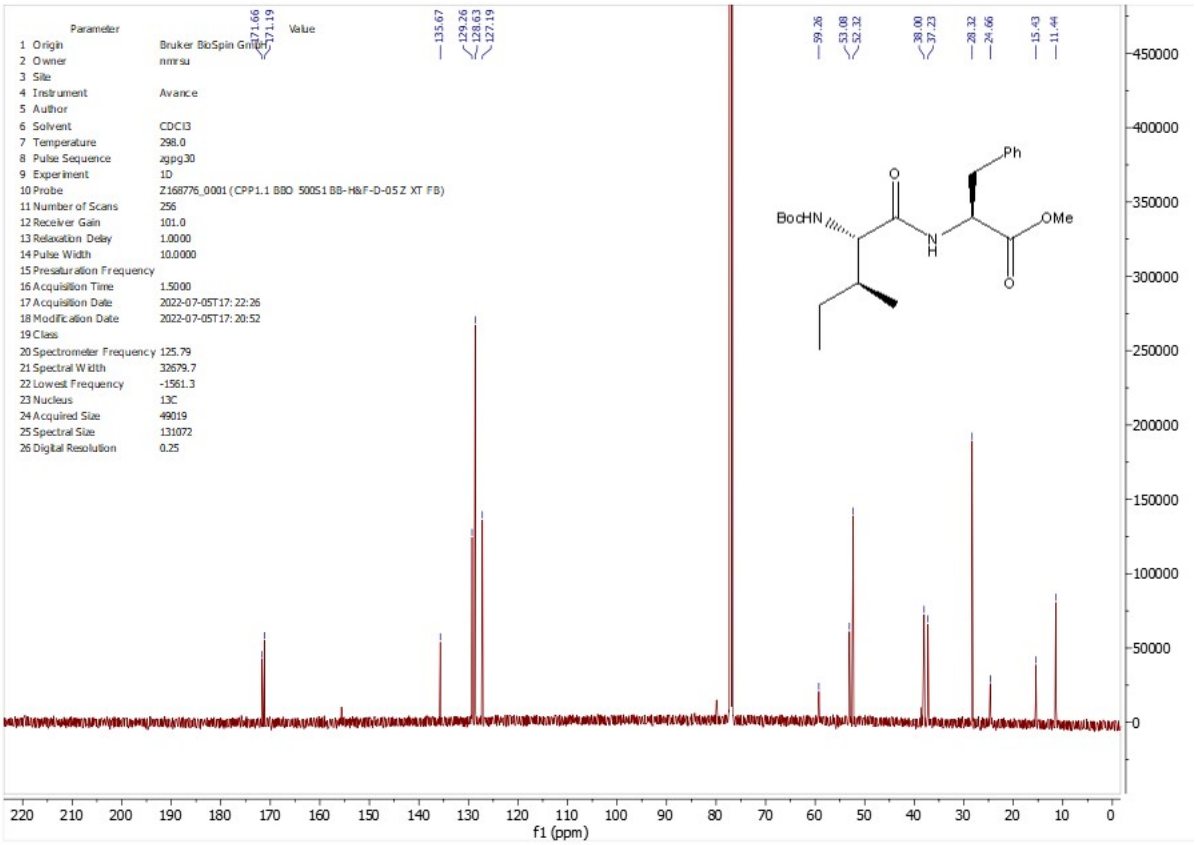
7 Temperature 298.0
8 Pulse Sequence zgpg30
9 Experiment 1D
10 Probe Z168776_0001 (CPP1.1 BBO 500S1 BB-H&F-D-05 Z XT FB)
11 Number of Scans 256
12 Receiver Gain 101.0
13 Relaxation Delay 1.0000
14 Pulse Width 10.0000
15 Presaturation Frequency
16 Acquisition Time 1.5000
17 Acquisition Date 2022-07-05T17:03:22
18 Modification Date 2022-07-05T17:01:48
19 Class
20 Spectrometer Frequency 125.79
21 Spectral Width 32679.7
22 Lowest Frequency -1561.3
23 Nucleus 13C
24 Acquired Size 49019
25 Spectral Size 131072
26 Digital Resolution 0.25
165.67
142.39
138.06
132.14
129.46
129.09
127.04
124.44
120.17
77.29
77.24
77.04
76.78
21.51
O
N
H
Me
f1 (ppm)

Boc-Ile-Phe-OMe (2)




Parameter Value
1 Origin Bruker BioSpin GmbH
2 Owner nmrsu
3 Site
4 Instrument Avance
5 Author
6 Solvent CDCl3
7 Temperature 298.0
8 Pulse Sequence zgpg30
9 Experiment 1D
10 Probe Z168776_0001 (CPP1.1 BBO 500S1 BB-H&F-D-05 Z XT FB)
11 Number of Scans 256
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13 Relaxation Delay 1.0000
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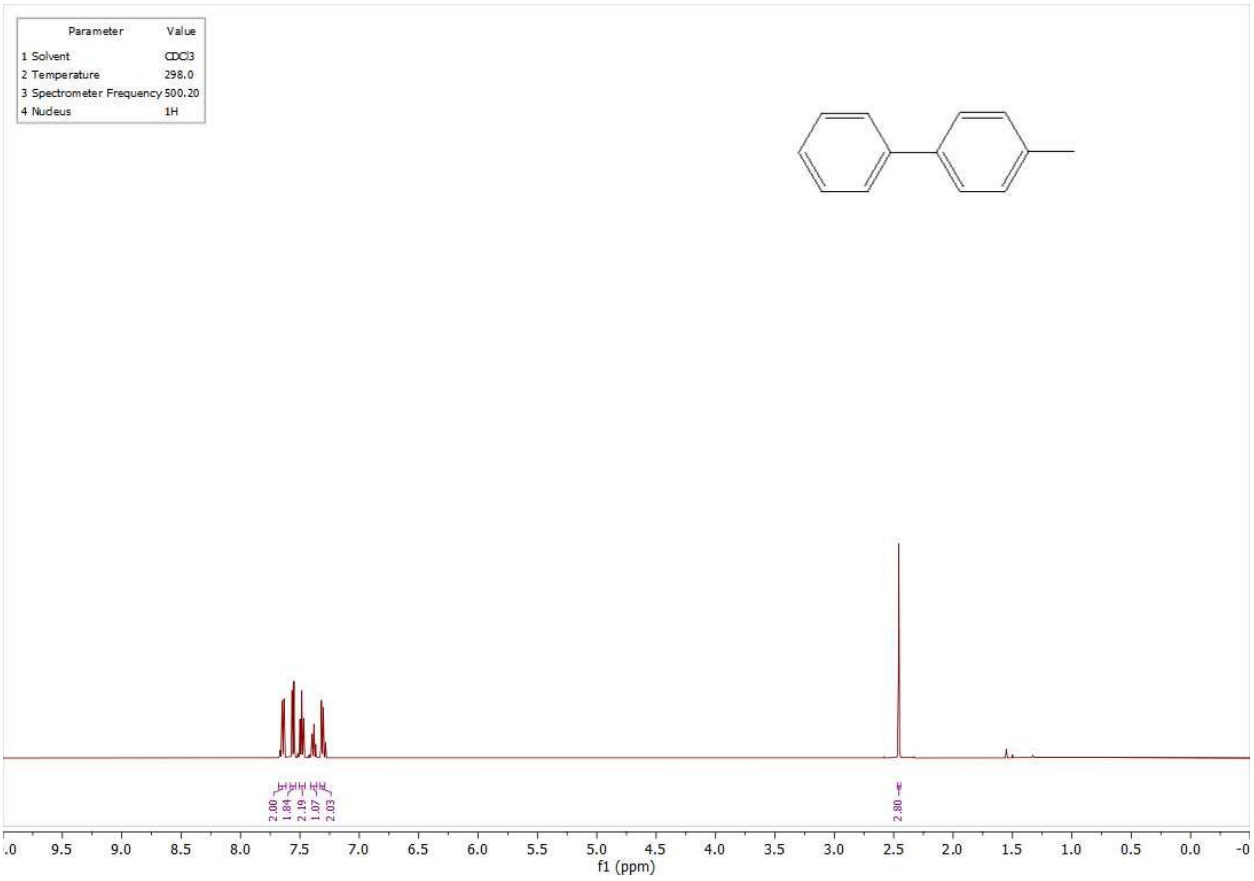
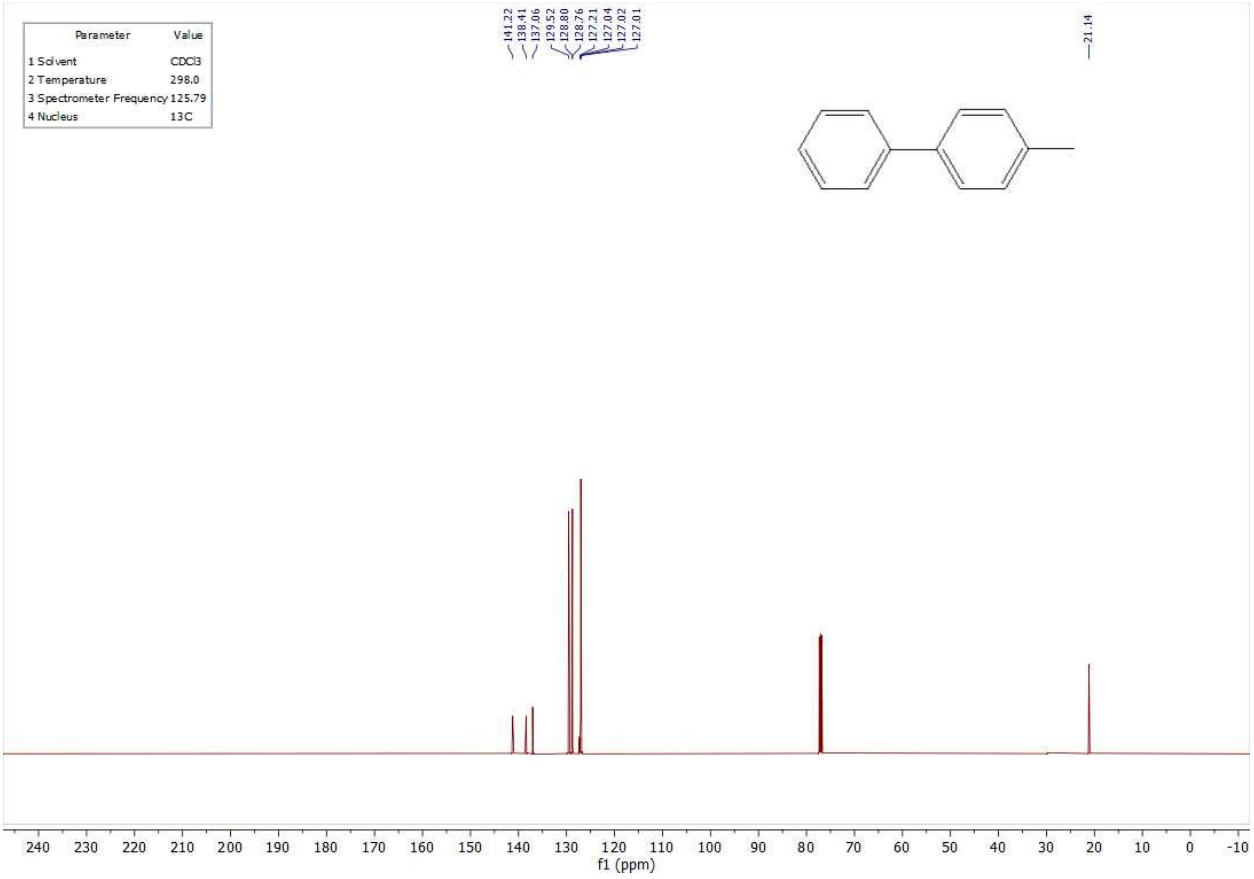
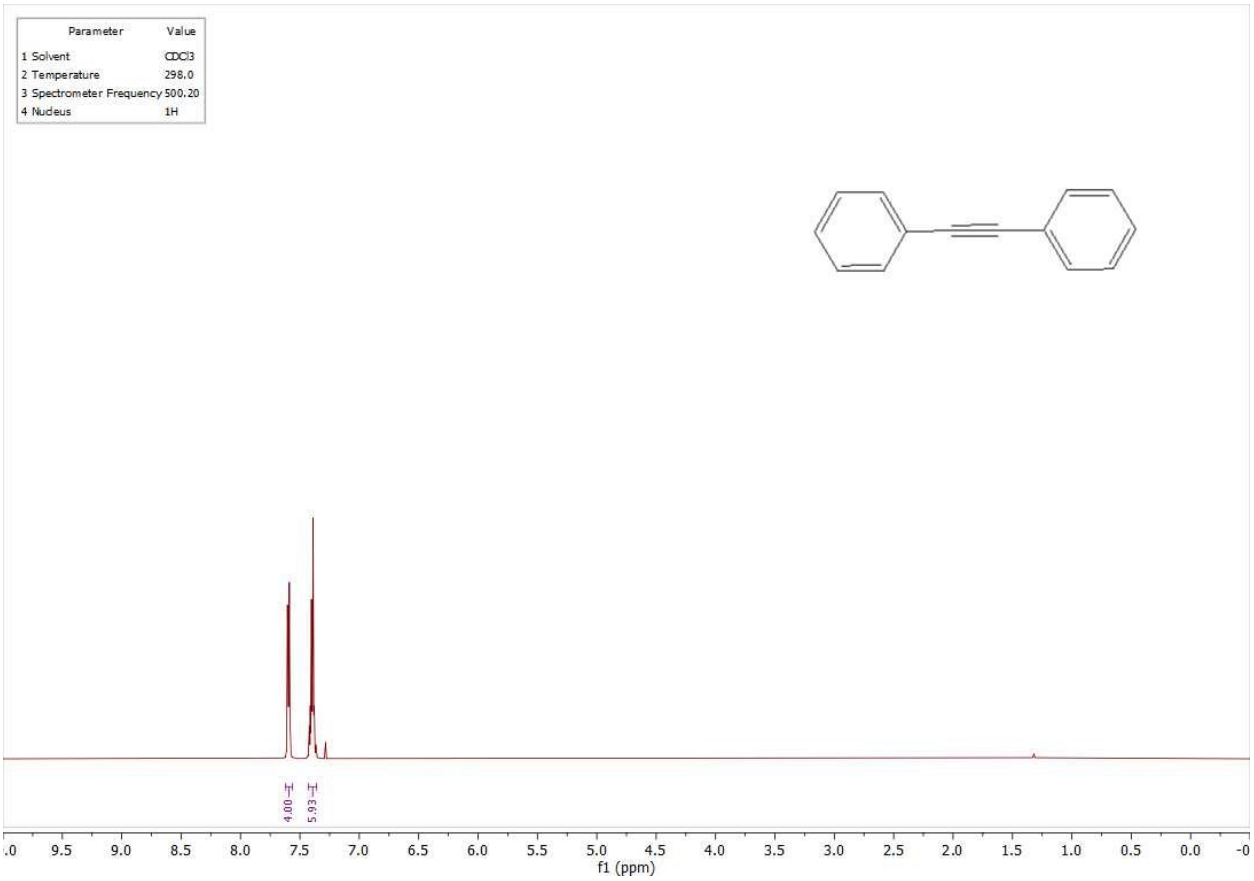
20 Spectrometer Frequency 125.79
21 Spectral Width 32679.7
22 Lowest Frequency -1561.3
23 Nucleus 13C
24 Acquired Size 49019
25 Spectral Size 131072
26 Digital Resolution 0.25
171.66
171.19
135.67
129.26
128.63
127.19
59.26
53.08
52.32
38.00
37.23
28.32
24.66
15.43
11.44
BocHN
Ph
OMe
N
H
O
O
O
450000
400000
350000
300000
250000
200000
150000
100000
50000
0
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

4-phenyltoluene (3)




Parameter Value
1 Solvent CDCl3
2 Temperature 298.0
3 Spectrometer Frequency 125.79
4 Nucleus 13C
141.22
138.41
137.06
129.52
128.80
128.76
127.21
127.04
127.02
127.01
21.14
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

Diphenylacetylene (4)



Parameter	Value
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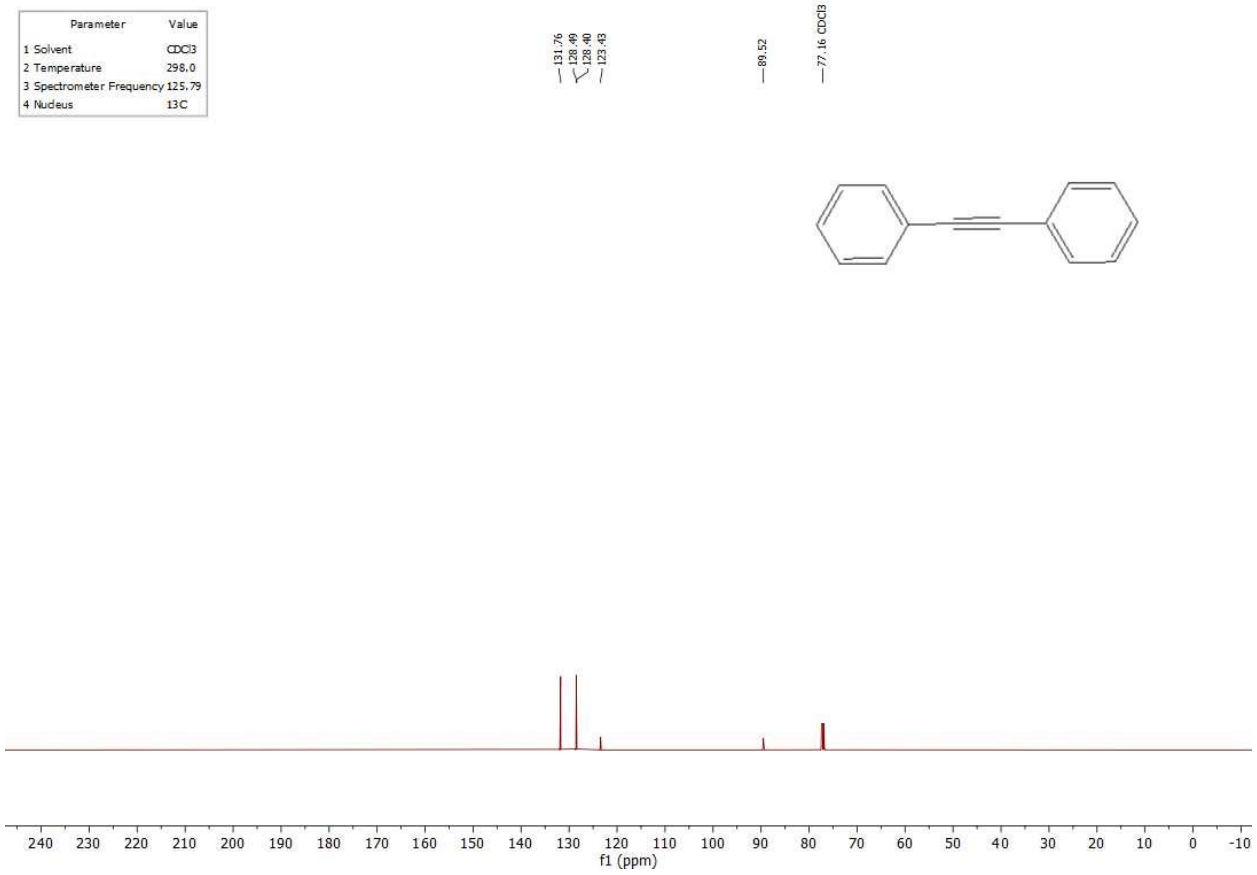
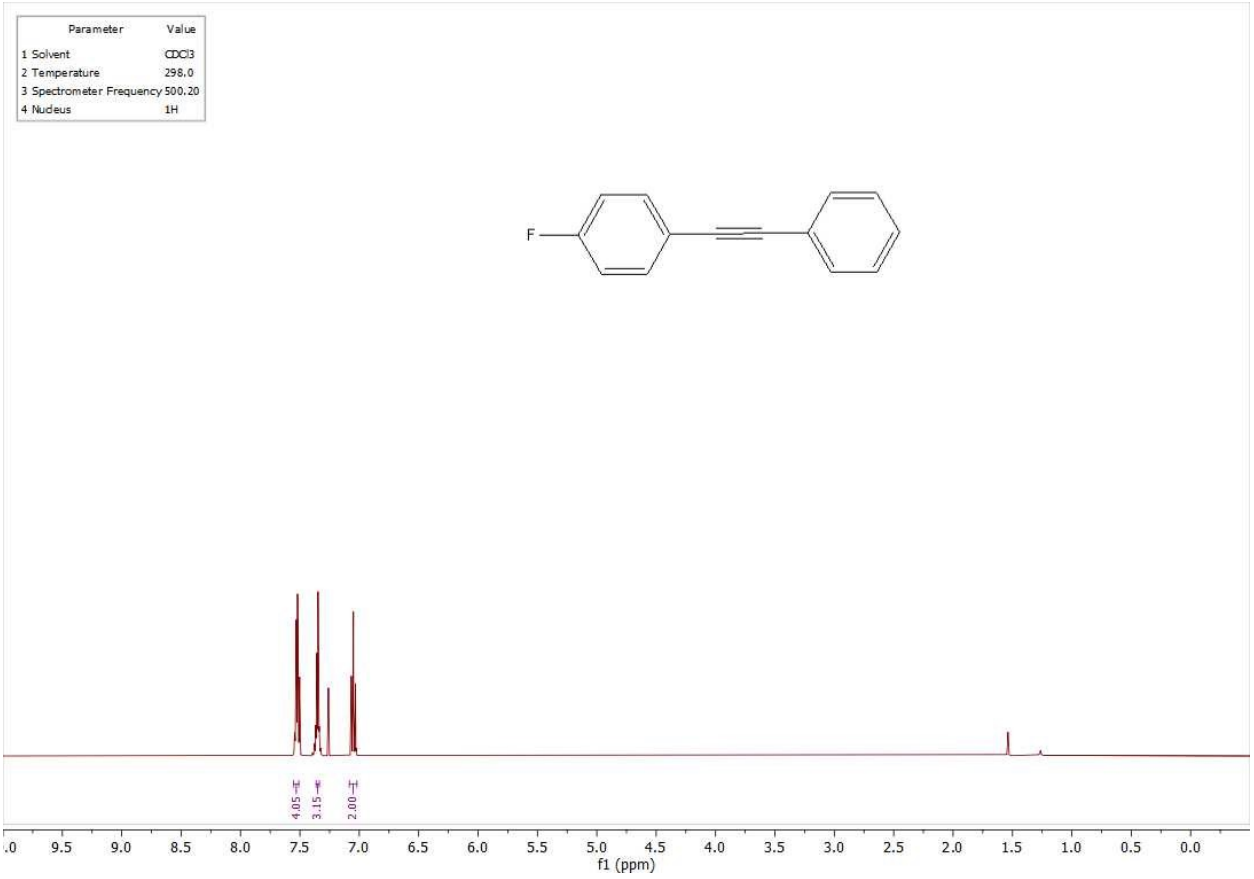
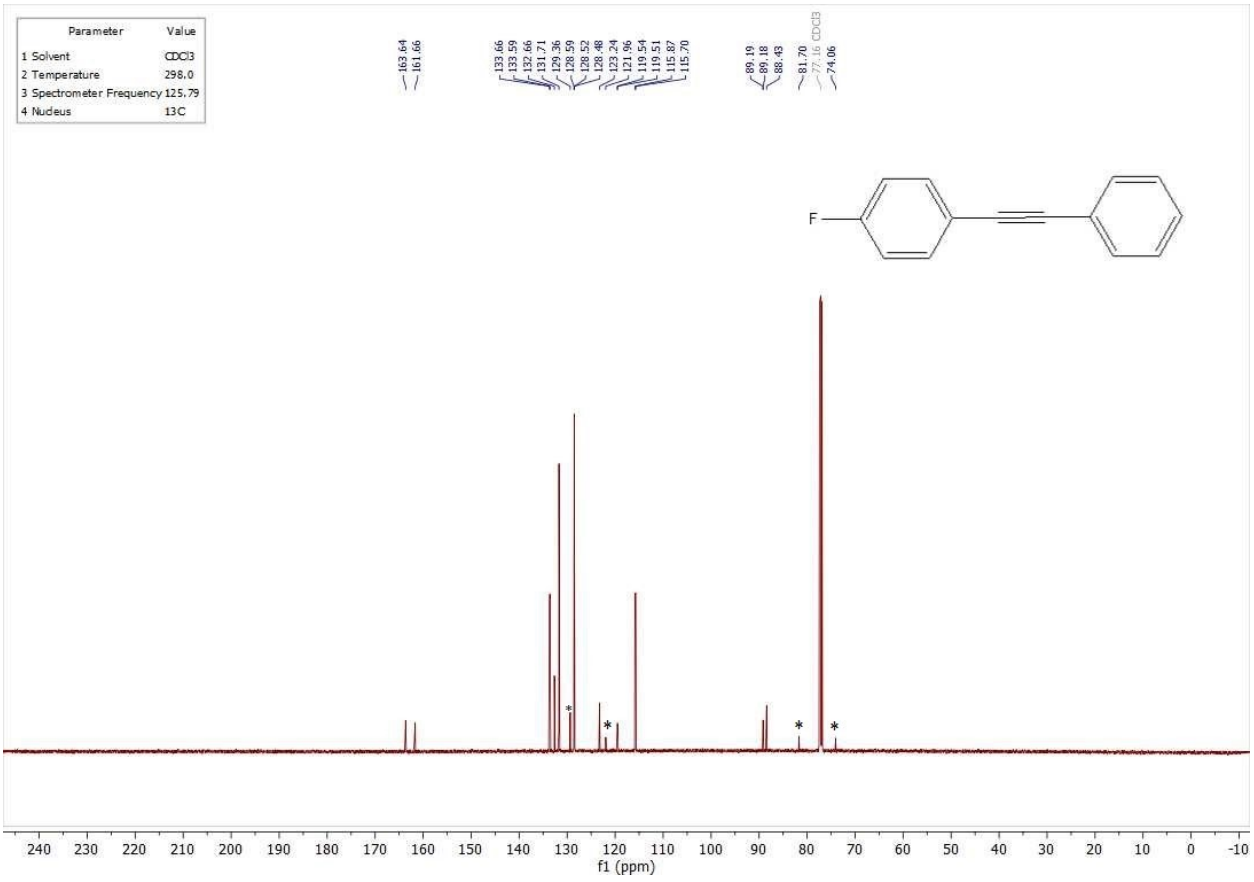
2 Temperature	298.0
3 Spectrometer Frequency	125.79
4 Nucleus	13C



1-fluoro-4-(phenylethynyl)benzene (5)

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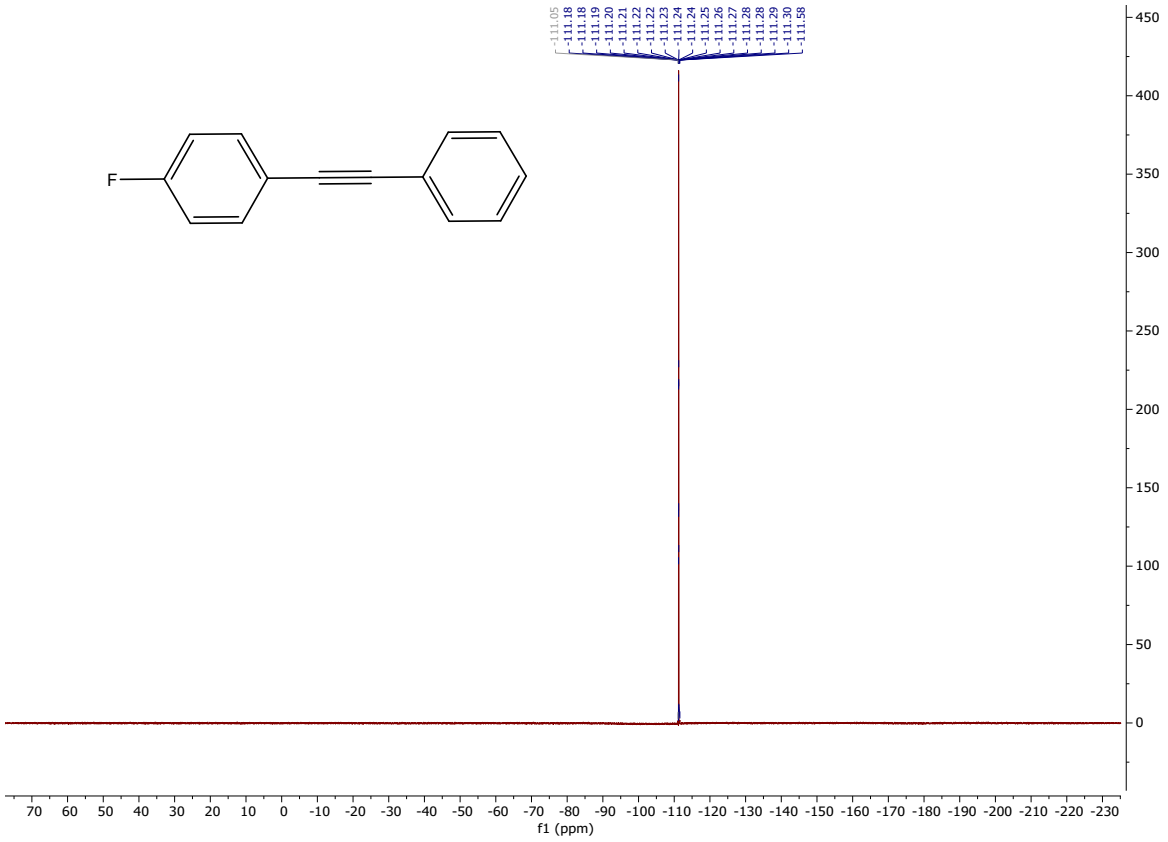
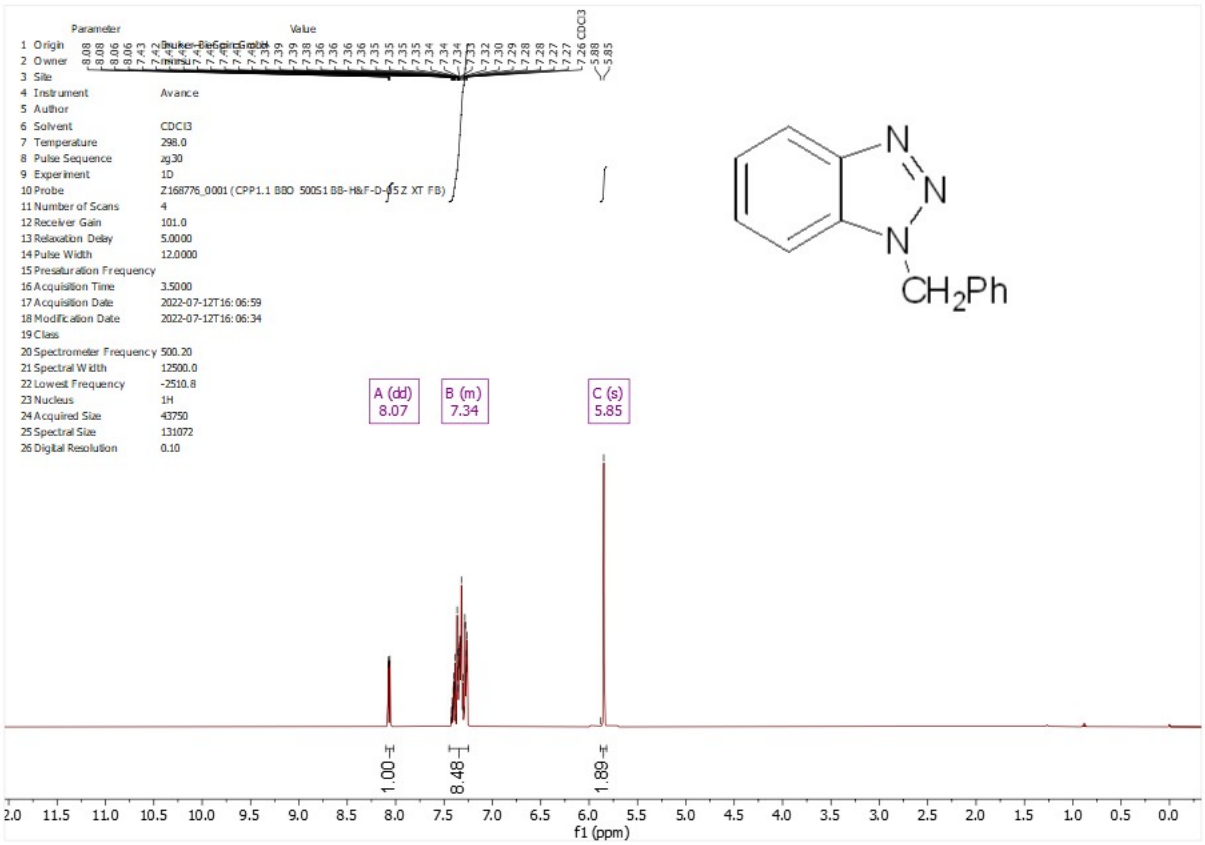
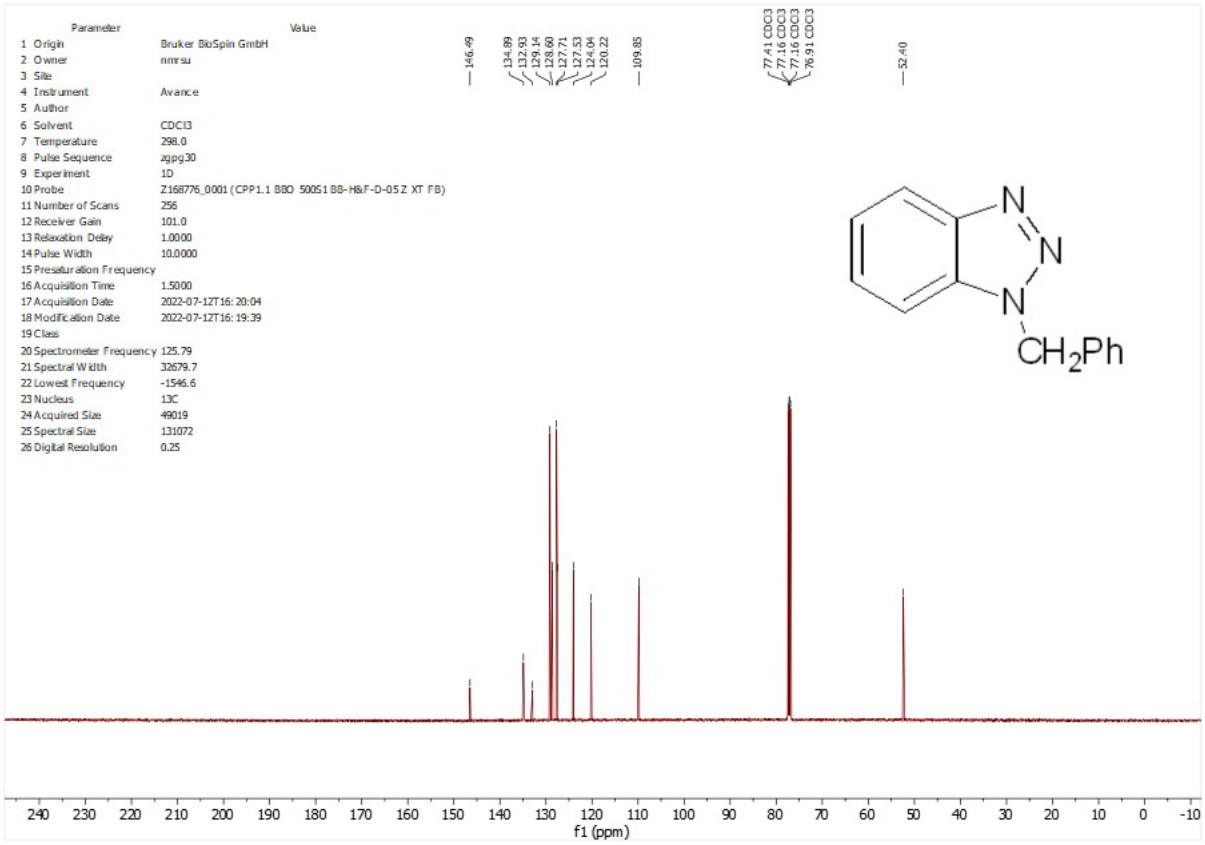
2 Temperature 298.0
3 Spectrometer Frequency 125.79
4 Nucleus 13C
163.64
161.66
133.66
133.59
132.66
131.71
129.36
128.59
128.52
128.48
123.24
121.96
119.54
119.51
115.87
115.70
89.19
89.18
88.43
81.70
77.16 CDCl3
74.06
F
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


-111.05
-111.18
-111.18
-111.19
-111.20
-111.21
-111.22
-111.22
-111.23
-111.24
-111.24
-111.25
-111.26
-111.27
-111.28
-111.28
-111.29
-111.30
-111.58
F
450
400
350
300
250
200
150
100
50
0
70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220 -230
f1 (ppm)

1-benzyl-1H-1,2,3-benzotriazole (6)



ff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2 Owner nmrsu
3 Site
4 Instrument Avance
5 Author
6 Solvent CDCl3
7 Temperature 298.0
8 Pulse Sequence zgpg30
9 Experiment 1D
10 Probe Z168776_0001 (CPP1.1 BBO 500S1 BB-H&F-D-05 Z XT FB)
11 Number of Scans 256
12 Receiver Gain 101.0
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14 Pulse Width 10.0000
15 Presaturation Frequency
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18 Modification Date 2022-07-12T16:19:39
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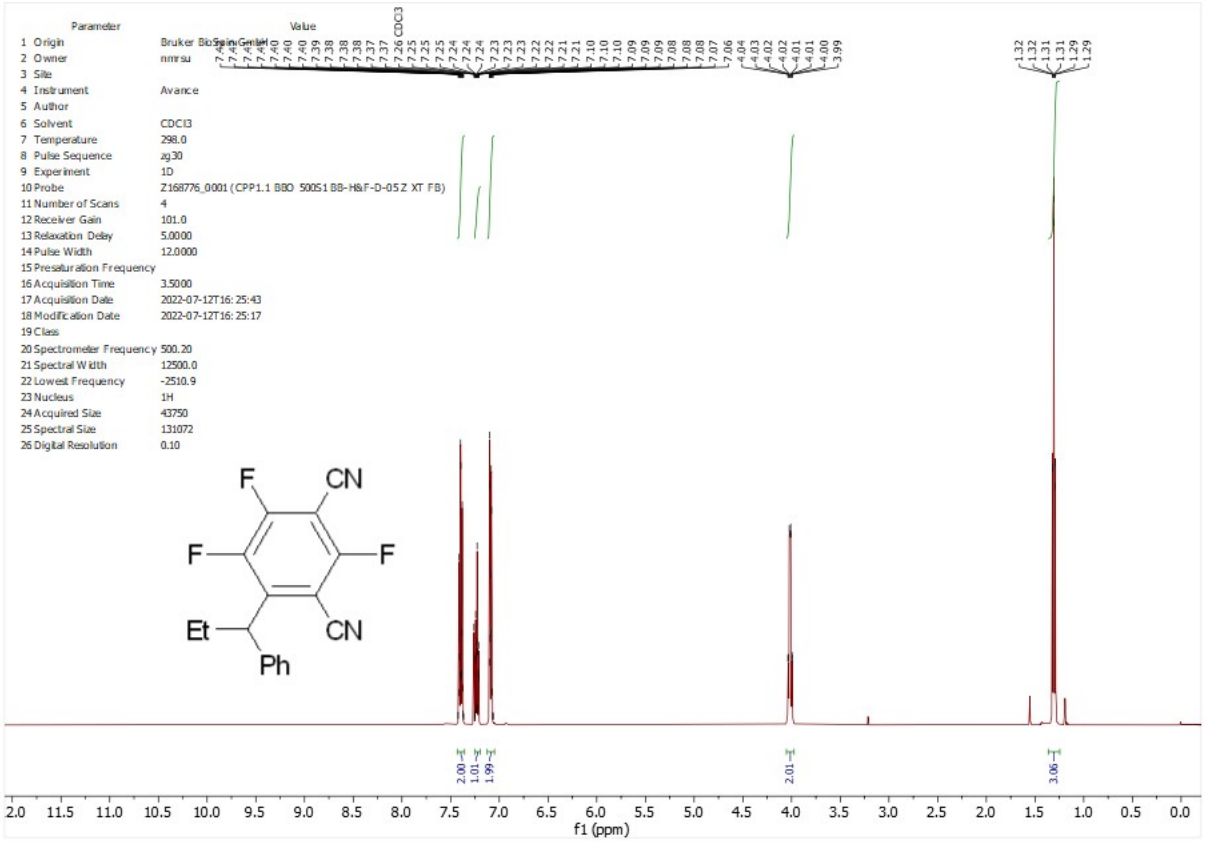
21 Spectral Width 32679.7
22 Lowest Frequency -1546.6
23 Nucleus 13C
24 Acquired Size 49019
25 Spectral Size 131072
26 Digital Resolution 0.25
146.49
134.89
132.93
129.14
128.60
127.71
127.53
124.04
120.22
109.85
77.41 CDCl3
77.16 CDCl3
77.16 CDCl3
76.91 CDCl3
52.40
N
N
N
CH2Ph
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

3FEPAIPN (7)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Value
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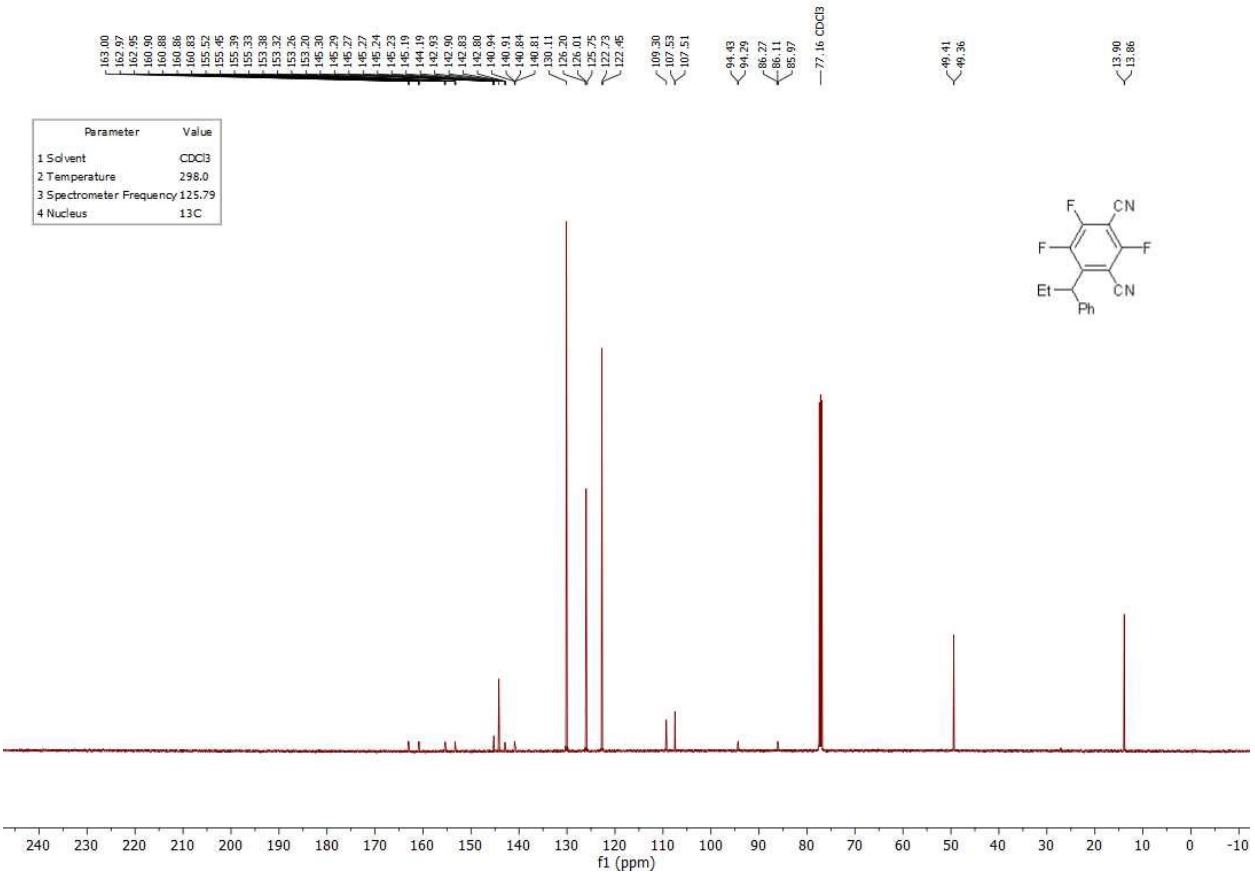
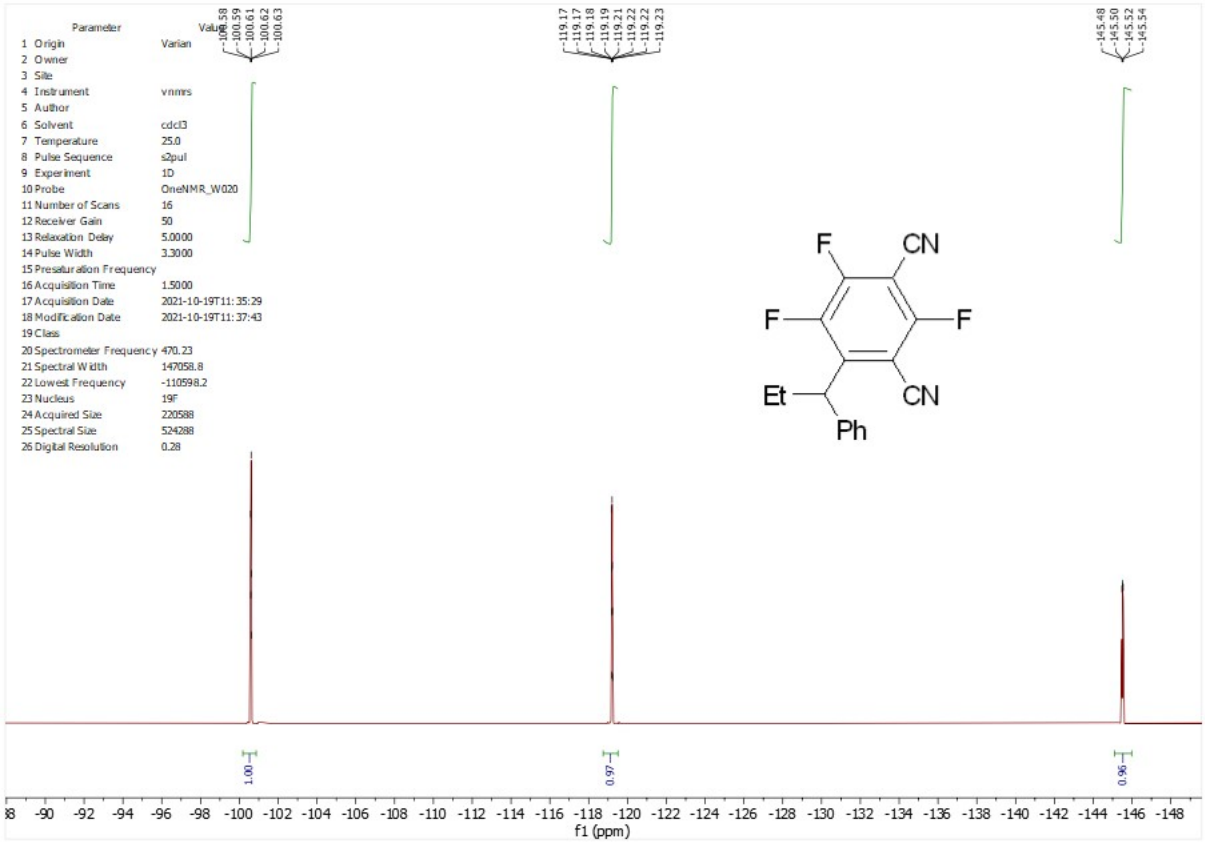
Bruker BioSpin GmbH
2 Owner
nmrsu
3 Site
4 Instrument
Avance
5 Author
6 Solvent
CDCl3
7 Temperature
298.0
8 Pulse Sequence
zg30
9 Experiment
1D
10 Probe
Z168776_0001 (CPP1.1 BBO 500S1 BB-H&F-D-05 Z XT FB)
11 Number of Scans
4
12 Receiver Gain
101.0
13 Relaxation Delay
5.0000
14 Pulse Width
12.0000
15 Presaturation Frequency
16 Acquisition Time
3.5000
17 Acquisition Date
2022-07-12T16:25:43
18 Modification Date
2022-07-12T16:25:17
19 Class
20 Spectrometer Frequency
500.20
21 Spectral Width
12500.0
22 Lowest Frequency
-2510.9
23 Nucleus
1H
24 Acquired Size
43750
25 Spectral Size
131072
26 Digital Resolution
0.10
F
CN
F
F
Et
CN
Ph
2.00
1.01
1.99
2.01
3.06
f1 (ppm)


163.00
162.97
162.95
160.90
160.88
160.86
160.83
155.52
155.45
155.39
155.33
153.38
153.32
153.26
153.20
145.30
145.29
145.27
145.27
145.24
145.23
145.19
144.19
142.93
142.90
142.83
142.80
140.94
140.91
140.84
140.81
130.11
126.20
126.01
125.75
122.73
122.45
109.30
107.53
107.51
94.43
94.29
86.27
86.11
85.97
77.16 CDCl3
49.41
49.36
13.90
13.86
Parameter Value
1 Solvent CDCl3
2 Temperature 298.0
3 Spectrometer Frequency 125.79
4 Nucleus 13C
F
CN
F
F
Et
CN
Ph
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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14 Pulse Width 3.3000
15 Presaturation Frequency
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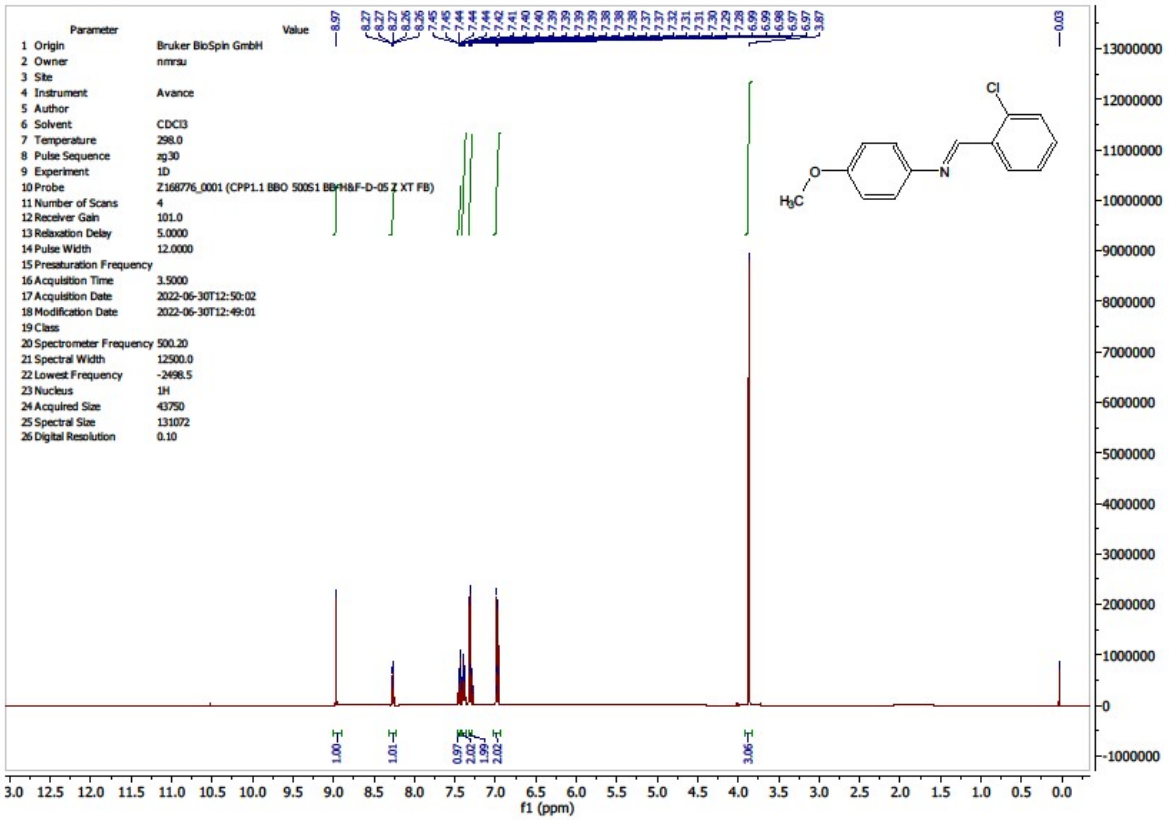
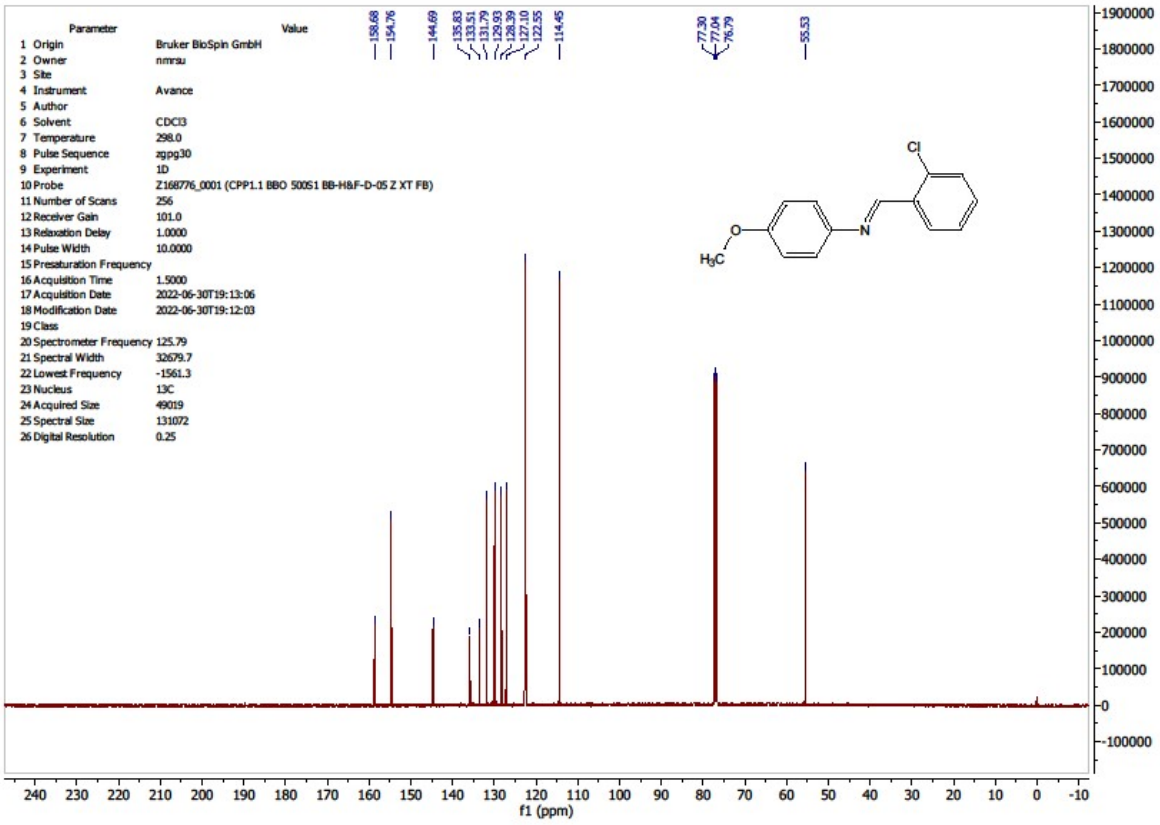
21 Spectral Width 147058.8
22 Lowest Frequency -110598.2
23 Nucleus 19F
24 Acquired Size 220588
25 Spectral Size 524288
26 Digital Resolution 0.28
-100.58
-100.59
-100.61
-100.62
-100.63
-119.17
-119.17
-119.18
-119.19
-119.21
-119.22
-119.22
-119.23
-145.48
-145.50
-145.52
-145.54
F
CN
F
F
Et
CN
Ph
1.00
0.97
0.96
-88 -90 -92 -94 -96 -98 -100 -102 -104 -106 -108 -110 -112 -114 -116 -118 -120 -122 -124 -126 -128 -130 -132 -134 -136 -138 -140 -142 -144 -146 -148
f1 (ppm)

N-[(2-chlorophenyl)methylene]-4-methoxy-benzenamine (8)




Parameter Value
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3 Site
4 Instrument Avance
5 Author
6 Solvent CDCl3
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8 Pulse Sequence zgpg30
9 Experiment 1D
10 Probe Z168776_0001 (CPP1.1 BBO 500S1 BB-H&F-D-05 Z XT FB)
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12 Receiver Gain 101.0
13 Relaxation Delay 1.0000
14 Pulse Width 10.0000
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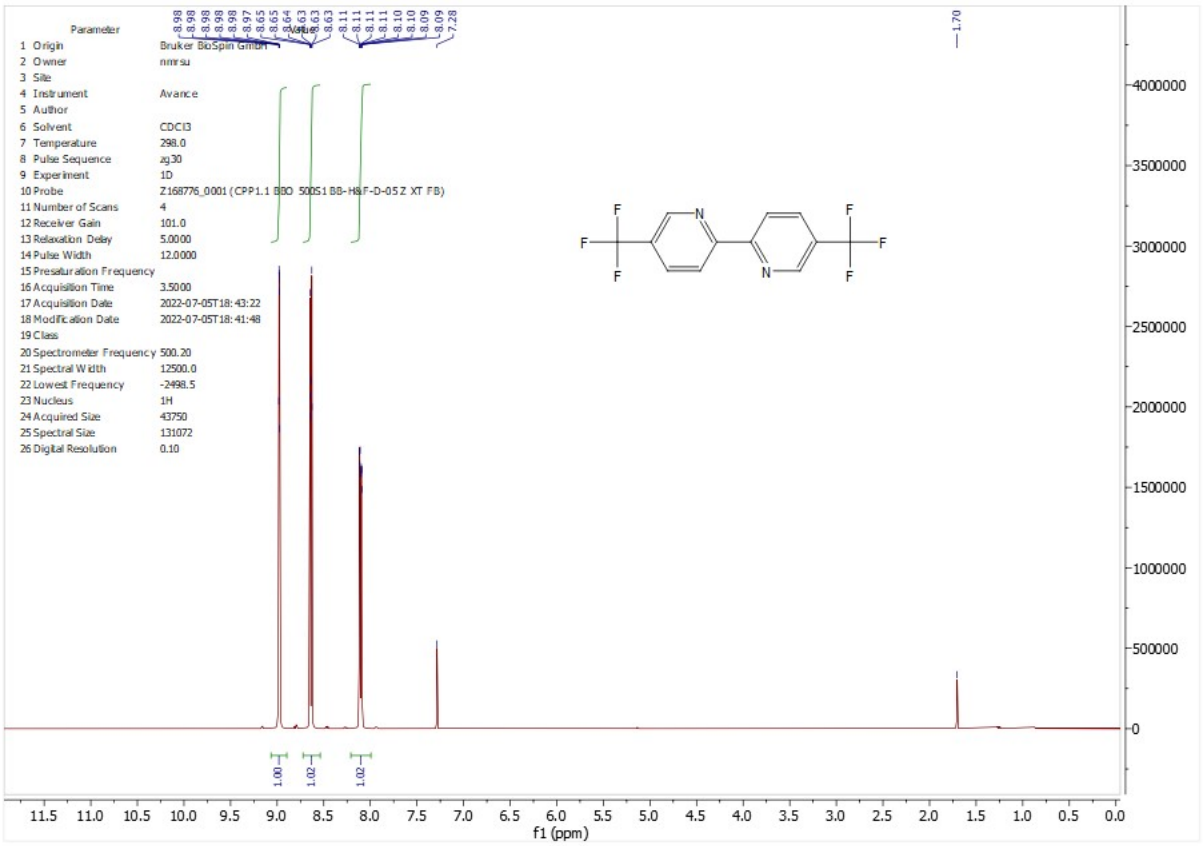
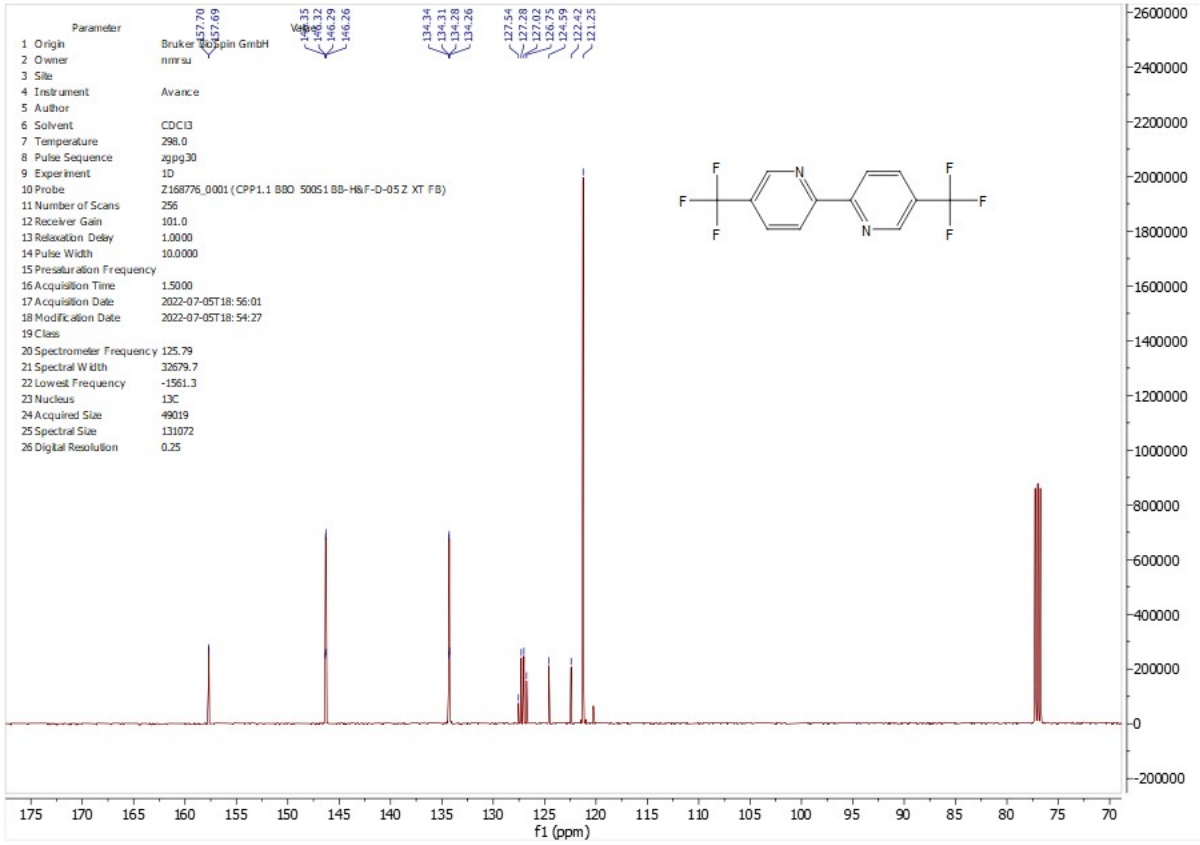
21 Spectral Width 32679.7
22 Lowest Frequency -1561.3
23 Nucleus 13C
24 Acquired Size 49019
25 Spectral Size 131072
26 Digital Resolution 0.25
158.68
154.76
144.69
135.83
133.51
131.79
129.93
128.39
127.10
122.55
114.45
77.30
77.04
76.79
55.53
Cl
O
N
H3C
f1 (ppm)

5,5’-Bis(trifluoromethyl)-2,2’-bipyridine




Parameter
1 Origin Bruker BioSpin GmbH
2 Owner nmrsu
3 Site
4 Instrument Avance
5 Author
6 Solvent CDCl3
7 Temperature 298.0
8 Pulse Sequence zgpg30
9 Experiment 1D
10 Probe Z168776_0001 (CPP1.1 BBO 500S1 BB-H&F-D-05 Z XT FB)
11 Number of Scans 256
12 Receiver Gain 101.0
13 Relaxation Delay 1.0000
14 Pulse Width 10.0000
15 Presaturation Frequency
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21 Spectral Width 32679.7
22 Lowest Frequency -1561.3
23 Nucleus 13C
24 Acquired Size 49019
25 Spectral Size 131072
26 Digital Resolution 0.25
157.70
157.69
146.35
146.32
146.29
146.26
134.34
134.31
134.28
134.26
127.54
127.28
127.02
126.75
124.59
122.42
121.25
F
N
F
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N
F
F
2600000
2400000
2200000
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1800000
1600000
1400000
1200000
1000000
800000
600000
400000
200000
0
-200000
175
170
165
160
155
150
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135
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115
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105
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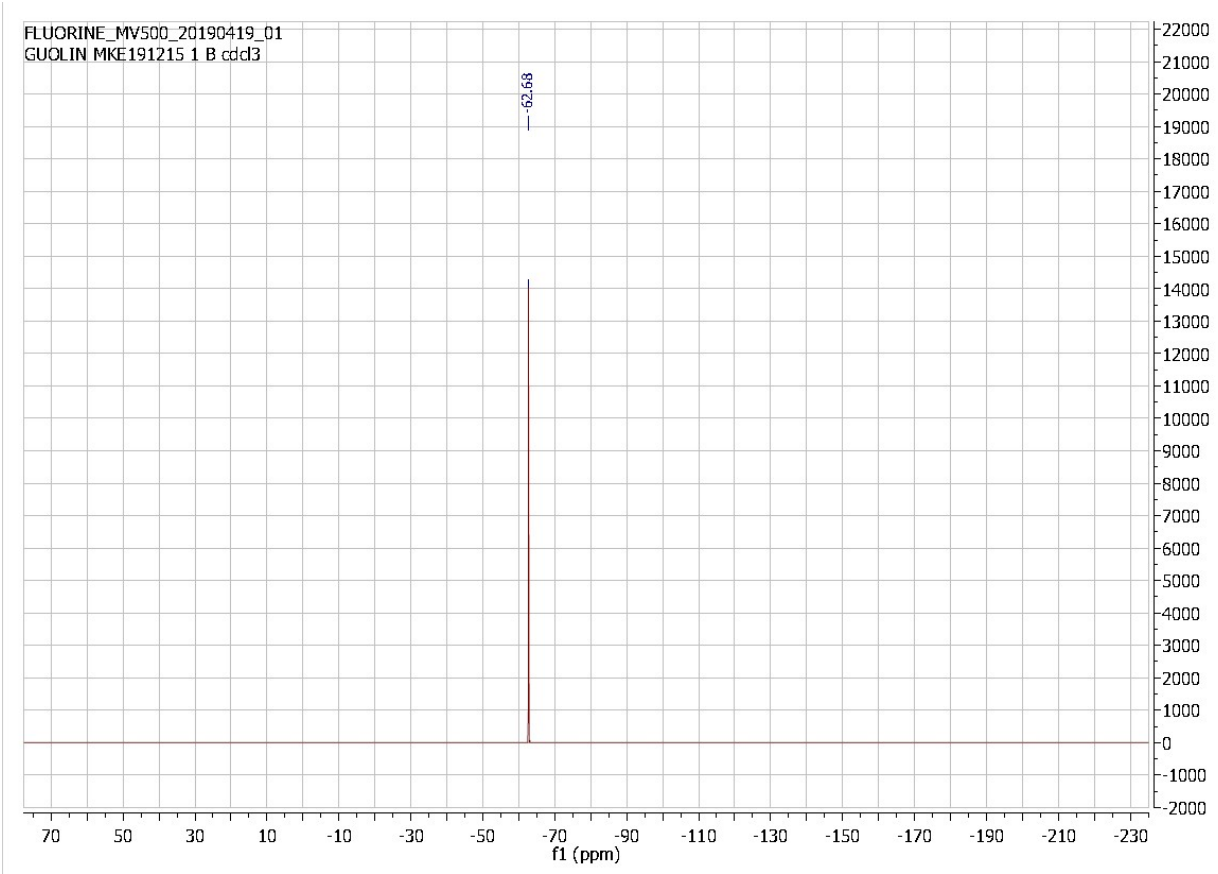
95
90
85
80
75
70
f1 (ppm)



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